

# CHNS Determination in reference soil samples

Reference methods:

**ISO 13878**: Soil quality - Determination of total nitrogen content by dry combustion (Elemental analysis) **ISO 15178**: Soil quality - Determination of total sulfur by dry combustion (Elementary analysis) Describes a procedure using dry combustion for the determination of total sulfur in soil samples. It is important to note that high temperature combustion methods might not determine total sulfur, only combustible sulfur. The difference between total and combustible sulfur is usually negligible in soils.

ISO 10694: Soil quality — Determination of organic and total carbon after dry combustion (Elemental analysis)
EN 13654-2: Soil improvers and growing media - Determination of nitrogen - Part 2: Dumas method
EN 13137: Characterisation of waste: determination of total organic carbon in waste, sludges and sediments.
ASTM D5291: Test method for Instrumental determination of C, H and N in petroleum products and lubricants.

Tested with VELP Scientifica EMA 502 Elemental Analyzer





## APPLICATION NOTE E-EMA-001-2021/A2

#### Introduction

A **carbon-to-nitrogen ratio (C:N ratio)** is a ratio of the mass of carbon to the mass of nitrogen in a substance. For example, if we have a C:N ratio of 24:1, this means we have 24 units of carbon to 1 unit of nitrogen. The C:N ratio is important because it has a direct impact on residue decomposition and also nitrogen cycling in soils.

It can, amongst other things, be used as indicator for nitrogen limitation of plants and other organisms. As a rule of thumb, the higher the ratio, the longer it takes for the material to decompose. Likewise, the smaller the ratio is, the more rapidly the plant material will decompose. This also has a direct relationship with the amount of nitrogen that is tied up in the soil that will be available to the next growing plant.

**Sulfur** is one of the main elements in the biosphere and an essential nutrient, it has some key functions in plants, like the formation of chlorophyll, that permits photosynthesis through which plants produce starch, sugars, oils, fats, vitamins and other compounds. So accurate and rapid determination of sulfur is important in soil and plant research and in agricultural monitoring programs.

**Hydrogen** is one of primary elements plants use in the largest amounts. The amount of hydrogen in the soil affects pH value too; best range for most nutrients to be available is from 6.0 to 7.0 and nutrient deficiencies can be observed at both high and low pH values.

#### **CHNS** determination in soils and sediments

The **CHNS** analysis starts with the combustion of the sample inside the VELP combustion furnace at a temperature higher than 1000°C to obtain elemental compounds.

**VELP Vcopper**<sup> $\mathbf{M}$ </sup>, a formulation of highly active copper powder, is placed in the lower part of the reactor helps the reduction of NO<sub>x</sub> into N<sub>2</sub>.

The gas stream reaches the gas-chromatographic column and flows out of the column after component separation with different retention times. The innovative  $LoGas^{TM}$  Thermal Conductivity Detector (TCD), with no requirement for a reference gas, enables the detection of all gases content.

The EMA 502 is controlled via PC through the intuitive **EMASoft**<sup>™</sup>. The analysis is completed in few minutes.



#### EMA 502 Preliminary Operations (daily)

Follow the operating manual to start the EMA 502 and check that the following parameters are set:

Temperature CHNS reactor (Code A00000443): 1030 °C Temperature GC Column Oven: 55 °C Flow rate MFC1 He: 120 ml/min Flow rate MFC2 He: 140 ml/min

Condition the system by testing 2 Sulphanilic acid standard (Code A00000434) and 2 to 3 empty tin foils (Code A00000436) as checkup. Verify the calibration curves with one or more tests as Standard by testing the same standard used for the curves creation.

#### **Sample Preparation**

Reference soil samples: Reference soil Expected range values declared on the certificate: C%=0.09 - 0.13% S%=0.09 - 0.13%

The sample has been analyzed as is.



### **Analysis Procedure**

Fill the following fields in the database: Sample name, Weight, Method, Sample type, Calibration number

Create a new customizable method with the following parameters:

O<sub>2</sub> flow rate: 300 ml/min O<sub>2</sub> factor: 0.7 ml/mg Min Oxy volume: 10 ml Press () to start the analysis. Analysis time: from 12 minutes for one run.

#### **Typical CHNS Results on soil samples**

**CHNS Calibration**: the CHNS results have been obtained with the calibration curve using the certified standard Sulphanilic acid (code A00000434) (C%= 41.58 - H% = 4.04 - N% = 8.08 - S% = 18.48), using a range of 0.15 - 0.5 mg of Sulphanilic acid.

The data obtained are included in the tolerance admitted by the certificate of analysis for Carbon and Sulphur content (declared parameters). The table below shows the %C, %S, %N and %H, obtained by the **EMASoft™** using 45-50 mg of sample.

Sample	%N (Average)	%C (Average)	%H (Average)	%S (Average)
Soil	0.0320	0.1247	0.3062	0.1173
	0.0309	0.1214	0.3148	0.1124
	0.0307	0.1276	0.3141	0.1170
	0.0314	0.1237	0.3102	0.1175
Average %	0.0312	0.1237	0.3113	0.1160
RSD %	1.68	1.79	1.10	1.83
Theoretical Value	N.A.	0.12	N.A.	0.11

#### Conclusion

The EMA 502 Elemental Analyzer CHNS-O is a reliable solution for the determination of carbon, hydrogen, nitrogen and sulfur in soil samples.

#### The main advantages of VELP CHNS-O elemental analyzer:

- Precision and reproducibility with the LOD of 0.001 mgN with Helium and the RSD of 0.2% (Sulphanilic acid)
- Low cost per analysis thanks to innovative technology and genuine consumables
- Fast set up of the instrument
- Easy maintenance
- Intuitive software User Interface
- Connectivity to VELP Ermes Cloud Platform for remote Application and Service support, remote access to the instruments, alerts and notifications and much more
- Software upgrade with the optional 21 CFR Part 11 Package for Pharmaceutical, Cosmetic and Food industry laboratories that require compliance with FDA regulation

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